



UNITED STATES PATENT AND TRADEMARK OFFICE

APPLICANT: GUIDOTTI ET AL. )  
)  
SERIAL NUMBER: 09/432,334 ) Group Art Unit: 1745  
)  
FILED: 11/2/99 )  
)  
FOR: ENERGY STORAGE AND )  
CONVERSION DEVICES USING ) Examiner: J. Maples  
THERMAL SPRAYED )  
ELECTRODES )

DECLARATION PURSUANT TO 37 C.F.R. § 1.132

The Assistant Commissioner of Patents  
Washington, D.C. 20231

Sir:

Tongsan D. Xiao, declares and says that:

1. I have worked in the field of thermally sprayed coatings for 10 years. I am an inventor of the above referenced patent application.

2. I have read, understood, and am familiar with the above-referenced application. I have also read, understood, and am familiar with the U.S. Patent No. 5,716,422 to Muffoletto et al.

3. During initial investigations into thermally spraying active materials that are thermally unstable at thermal spray temperatures, attempts were made to form an FeS<sub>2</sub> (pyrite) coating by thermally spraying FeS<sub>2</sub> as described by Muffoletto. First attempts employed high-velocity oxygen flame (HVOF). These attempts were unsuccessful in that

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FeS<sub>2</sub> was not deposited. The deposited material was analyzed by X-ray diffraction (XRD) and was found to be primarily Fe<sub>2</sub>O<sub>3</sub>. Further attempts to obtain an FeS<sub>2</sub> coating employed dc-arc plasma spray under an argon cover (250 SCFH) with arc currents of between 200 and 300 A. XRD of the deposited material indicated Fe<sub>7</sub>S<sub>8</sub> and not FeS<sub>2</sub>. Representative data is shown in Fig. 1 attached hereto.

4. FeS<sub>2</sub> was plasma sprayed with elemental sulfur. The primary phase of the resulting deposit (>95%) was FeS<sub>2</sub> as shown by the X-ray data in Figure 1.

5. Discharge traces were recorded for cells employing natural FeS<sub>2</sub>, FeS<sub>2</sub> plasma sprayed without sulfur and FeS<sub>2</sub> plasma sprayed with sulfur. These discharge traces are shown in Fig. 2 attached hereto. The discharge traces corroborate the above described XRD data in that the FeS<sub>2</sub> plasma sprayed without sulfur exhibits a significantly different trace than the natural pyrite and the FeS<sub>2</sub> plasma sprayed with sulfur. Additionally the discharge traces of the cell employing FeS<sub>2</sub> plasma sprayed with sulfur paralleled the discharge traces of the cell employing natural pyrite.

6. Thus, based on my knowledge of the thermal spray field and the above described experiments and data, I believe the presence of a protective coating such as elemental sulfur is to be necessary to produce a thermally sprayed electrode comprising  $\text{FeS}_2$  or other material that is unstable at thermal spray temperatures.

7. I declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or document or any patent resulting therefrom.

Date: \_\_\_\_\_

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Tongsan D. Xiao